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(54) LOW-MAGNETIC BLACK PIGMENT POWDER, ITS PRODUCTION METHOD AND ITS USE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a production method for a low magnetic black pigment powder, displaying such as blackness as a magnetite and having no sintering and agglomeration of particles.

SOLUTION: The low magnetic black pigment powder is a mixed-phase crystalline, having a rutile-type TiO₂ phase as a base, wherein the base is characterized by a particle structure covered with Fe₂TiO₄ phase, and having a peak of Fe₂TiO₄ with the rutile-type TiO₂ as a main peak in X-ray diffraction. A toner composition, a paint composition, a resin composition, a cosmetic and the like are produced by using the low magnetic black pigment powder.

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CLAIMS

[Claim(s)]

[Claim 1] Low magnetism black pigment powder which uses rutile mold TiO_2 phase as a base, and is characterized by the particulate structure by which this base was covered with Fe_2TiO_4 phase.

[Claim 2] Low magnetism black pigment powder according to claim 1 characterized by being a mixed phase crystal including the peak of Fe_2TiO_4 by making the rutile mold TiO_2 into MEIMPI-KU in an X diffraction.

[Claim 3] Low magnetism black pigment powder according to claim 1 or 2 characterized by for particle size being 0.1-0.4 micrometers, and a magnetization value being 0.5 - 10.0 emu/g. $(= \text{Am}^2/\text{kg})$

[Claim 4] Low magnetism black pigment powder according to claim 1 to 3 characterized by covering a kind or two sorts or more of inorganic oxides, water or anhydrous, chosen from the group which consists of silicon, aluminum, titanium, a zirconium, and tin on the Fe_2TiO_4 above-mentioned phase.

[Claim 5] Low magnetism black pigment powder according to claim 1 to 4 characterized by performing hydrophobing processing by the silicone oil and/or the coupling agent on the Fe_2TiO_4 above-mentioned phase.

[Claim 6] The manufacture approach of the low magnetism black pigment powder characterized by returning further and forming Fe_2TiO_4 phase after covering with the 30 - 120 weight section to titanium oxide at the hydrous or anhydrous titanium oxide which is a base by setting iron salt to Fe_2O_3 , calcinating in a 700-1100-degree C oxidizing atmosphere and forming Fe_2TiO_5 phase on the surface of a base.

[Claim 7] The manufacture approach of the low magnetism black pigment powder according to claim 6 characterized by using for a base the hydrous titanium oxide which has the specific surface area more than $200\text{m}^2/\text{g}$.

[Claim 8] The manufacture approach of the low magnetism black pigment powder according to claim 6 or 7 characterized by returning at the temperature of 400-550 degrees C, using hydrogen gas and the mixed gas of carbon dioxide gas as a reducing agent.

[Claim 9] The toner constituent characterized by using low magnetism black pigment powder according to claim 1 to 5.

[Claim 10] The coating characterized by using low magnetism black pigment powder according to claim 1 to 5.

[Claim 11] The resin constituent characterized by using low magnetism black pigment powder according to claim 1 to 5.

[Claim 12] The charge of makeup characterized by using low magnetism black pigment powder according to claim 1 to 5.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] Dispersibility of this invention is good as coloring agents, such as resin including a coating, and a charge of makeup, in an environmental list as a coloring agent of the toner which is a developer for electrophotography in more detail about insurance and the black pigment powder of low magnetism which is harmless and does not have magnetic condensation to the body, and high black pigment powder whenever black is offered.

[0002]

[Description of the Prior Art] The one component system development method developed using the toner itself which distributed the coloring agent in binder resin to the development method in electrophotography, and a toner and a carrier are mixed, and it can divide roughly into the binary system development method which conveys a toner with a carrier and is developed.

[0003] It faces copying in any case, the electrostatic latent image formed in the photo conductor is developed with these toners, the toner powder which carried out image formation on the photo conductor is imprinted to imprint material, such as paper and a sheet, it is established using heat, a pressure, etc., and a permanent image is obtained. In these toners, carbon black and magnetite which are a black coloring agent as image formation material are used so much. Among these, carbon black is used in a binary system development method or a nonmagnetic 1 component development method, and magnetite is used for the magnetic 1 component development method which conveys a toner with the MAG.

[0004] The amount of these coloring agents used tends to increase further with the increment in need of the toner as an image formation ingredient accompanying the advance of the latest computer.

[0005] In recent years, it comes to talk about the problem of environmental pollution or a health hazard greatly, and the demand about the safety of a use raw material is increasing also in the toner.

[0006] The aromatic hydrocarbon of ultralow volume contains in the carbon black currently used so much as a black coloring agent of a toner, and the trend to regard safety as questionable about the toner using carbon black is growing into this from the matter which is having carcinogenic [like 3,4-Benzo-pyren] pointed out containing.

[0007] On the other hand, although magnetite is a black pigment which uses insurance and a harmless iron oxide as a principal component, since it has high magnetism, particles re-condense and there is a problem that a uniform dispersing element is difficult to get. Moreover, since magnetite has conductive ability, it cannot be used as a coloring agent of the toner of the above-mentioned 2 component development method which needs insulation or high resistance, or a nonmagnetic 1 component development method.

[0008] The black pigment particle powder which becomes JP,3-2276,A from the polycrystal particle which has a mixed presentation with the Fe₂TiO₅ and Fe₂O₃-FeTiO₃ solid solution as a nonmagnetic black pigment which replaces carbon black, and the black particle powder which has the hematite structure containing Mn in JP,8-143316,A and JP,2000-10344,A are indicated.

[0009] A black pigment given in JP,3-2276,A covers titanium oxide to magnetite, or magnetite and titanium oxide are mixed. It is a thing about the black pigment particle powder which consists of a polycrystal particle which has a mixed presentation with the Fe_2TiO_5 and $\text{Fe}_2\text{O}_3\text{-FeTiO}_3$ solid solution calcinated and obtained by the non-oxidizing atmosphere. Although it does not become a problem about safety since insurance, and harmless ferrous oxide and titanium oxide are used as a principal component Since it is what is calcinating the magnetite which used strong coherent magnetite as the main raw material, and covered titanium oxide by high temperature 700 degrees C or more, It was what has a problem in that hematite generates, a color tone becomes redness, whenever [black] runs short, and only the particle which sintering or coalescence-izing between particles produced further is obtained, but mono dispersion is carried out to homogeneity. Moreover, since Mn which is an environmental pollutant is contained so much in a black pigment given in JP,8-143316,A and JP,2000-10344,A, it is hard to call it insurance and a harmless pigment.

[0010]

[Problem(s) to be Solved by the Invention] Thus, to the body, insurance and the black pigment with good dispersibility are unprecedented in the environmental list replaced with the carbon black currently used so much as a coloring agent of the toner of a 2 component development method or a nonmagnetic 1 component development method, and it is going to just look forward to the development harmless. Moreover, it can be used also as a coloring agent of business, such as resin, charges of makeup, etc. not only including a toner but a coating.

[0011]

[The means for solving a technical problem and the gestalt of implementation of invention] From such a situation, this invention persons are the multiple oxides which used the iron oxide as the principal component to the body in insurance and a harmless titanium oxide list at the environmental list, and repeated examination wholeheartedly that a black pigment with good dispersibility without sintering or coalescence-izing between particles should be developed. As a representative of the oxide which presents the black which used titanium oxide and an iron oxide as the principal component, there are FeTiO_3 (ilmenite) and Fe_2TiO_4 (Iron Titanium Spinel).

[0012] When this invention persons use titanium oxide as a base, and make Fe_2TiO_5 (shoe DOBURUKKAITO) form in the front face as a result of inquiring paying attention to Fe_2TiO_4 and it was returned, they found out that the titanium oxide covered with Fe_2TiO_4 was obtained, and completed this invention. As a result of measuring this product by the X diffraction, TiO_2 (rutile type titanium dioxide) was made into MEIMPI-KU, and having the peak of the multiple oxide of the various titanium containing Fe_2TiO_4 and iron was confirmed.

[0013] That is, the low magnetism black pigment powder of this invention uses rutile mold TiO_2 phase as a base, and is characterized by the particulate structure by which this base was covered with Fe_2TiO_4 phase. Moreover, this pigment powder can be specified as a mixed phase crystal including the peak of Fe_2TiO_4 by making the rutile mold TiO_2 into MEIMPI-KU in an X-ray diffraction method.

[0014] Although "the mixed phase crystal" said to this application means the polycrystal with which two or more single crystals generally exist, in the black pigment powder in connection with this invention, the condition that the multiple oxide phase which used as the principal component Fe_2TiO_4 phase uniform to extent which can be specified by the X diffraction was formed in the surface section which used titanium oxide as the base is said. At this point, it is distinguished from the conventional particle which generated a mixed presentation and hematite with the solid solution at least.

[0015] And whenever [equivalent to magnetite / black] was shown, it was a particle without sintering or coalescence-izing of a particle, particle size was 0.1-0.4 micrometers, and a magnetization value hardly had magnetism with 0.5 - 10.0 emu/g, but this black pigment powder was low magnetism powder, and was very good as a substitute of carbon black.

[0016] Furthermore, although the black pigment powder of this invention is usable as a coloring agent for toners even when it remains as it is An electrification nature [in / according to the need / to a heat-resistant list / improvement in concordance with resin, or a toner], and environmental stability amelioration sake, On the Fe_2TiO_4 above-mentioned phase, silicon, aluminum, titanium, A kind or two

sorts or more of inorganic oxides, water or anhydrous, chosen from the group which consists of a zirconium and tin may be covered, and performing hydrophobing processing by the silicone oil and/or the coupling agent may combine surface treatment processing of these various kinds with arbitration preferably.

[0017] The black multiple oxide which constitutes the black pigment powder of this invention can be obtained as follows. Namely, the manufacture approach of this invention uses hydrous or anhydrous titanium oxide as a base. After adding iron salt and covering with the 30 - 120 weight section as Fe_2O_3 , calcinate in a 700-1100-degree C oxidizing quality ambient atmosphere, and Fe_2TiO_5 uniform phase is made to form in a titanium oxide front face further. Subsequently It is characterized by returning the multiple oxide, returning Fe_2TiO_5 phase covered on the front face, and making it permute by Fe_2TiO_4 .

[0018] In the process of this invention, in order to obtain the above-mentioned black multiple oxide, it is important for a titanium oxide front face to form Fe_2TiO_5 uniform phase first, and it is required to cover to homogeneity the iron hydroxide or iron oxide obtained by for that to use a reactant good thing as a raw material of titanium oxide and the list by hydrolyzing a water-soluble iron compound on that front face.

[0019] As titanium oxide which can be used in this invention, the thing more than 20m²/g has a desirable specific surface area, especially hydrous hydroxylation titanium has a specific surface area as high as 200m²/g, and it is desirable especially from the point of being rich in reactivity. In addition, since the amount of [which is contained in water and titanium oxide] sulfuric acid influences and hematite generates at the time of baking in using for a raw material the water and titanium oxide obtained by the sulfuric-acid method, it is required to remove a part for a sulfuric acid by washing beforehand.

[0020] As water-soluble iron salt used in order to carry out the coat of Fe_2TiO_5 Ferrous chloride, a ferric chloride, a ferrous sulfate, ferric sulfate, the first iron of a nitric acid, the second iron of a nitric acid, etc. can be used. Moreover, although may neutralize as the approach of hydrolysis, it may oxidize with the approach of depositing, there may be the approach of depositing and any are sufficient, a coat with the more uniform approach of neutralizing and depositing is obtained, and since reactivity is also good, it is desirable. The amount of covering of an iron hydroxide or ferrous oxide has the desirable 30 - 120 weight section to titanium oxide. In below 30 weight sections, when it is more than the 120 weight sections, in order not to obtain the target black oxide, and for reduction to stop being able to progress easily and to obtain a high thing whenever black, since time amount is taken, it is not desirable.

[0021] And although it calcinates on it and Fe_2TiO_5 phase is made to form in it after covering an iron hydroxide or an iron oxide on the titanium oxide front face of a base, 700-1100-degree C 800-1000 degrees C are preferably suitable for the burning temperature at that time. Since the reaction of Fe_2TiO_5 phase will not be completed, but hematite will remain, and the magnetization value of the black multiple oxide obtained by returning becomes high and stability worsens when lower than 700 degrees C, it is not desirable. When it calcinates at the temperature of 1100 degrees C or more, the black multiple oxide of this invention can be obtained, but since primary particle size becomes larger than 0.4 micrometers and dispersibility worsens, it becomes unsuitable as a coloring agent for toners and is not desirable.

[0022] Next, Fe_2TiO_5 phase obtained by the above-mentioned baking is returned. When returning the titanium oxide with which Fe_2TiO_5 phase was covered by homogeneity, the solid-state reducing agent containing reducibility gas and carbon, such as hydrogen gas, carbon dioxide gas, carbon monoxide gas, ammonia gas, and amine gas, can be used, but when using the solid-state reducing agent containing carbon, it is desirable to make it carbon black not remain. In this invention, especially since Fe_2TiO_4 which using together hydrogen gas and carbon dioxide gas, and returning generates at the time of reduction is stabilized and is obtained, it is desirable. Although reduction can be performed at 250-700 degrees C, it is required to carry out with optimum temperature with the reducing agent to be used. When using together hydrogen gas and carbon dioxide gas and returning, 400-550 degrees C is the optimal. Since reduction does not progress, but generation of Fe_2TiO_4 becomes inadequate and whenever [black] falls when lower than 400 degrees C, it is not desirable. Moreover, if it is made higher than 550 degrees C, since sintering of a particle will take place and dispersibility will worsen at

the time of reduction, it is not desirable.

[0023] In the process of this invention, as occasion demands, if well-known sintering inhibitors, such as aluminum, silicon, Lynn, and a zirconium salt, are used, the black multiple oxide which was further excellent in dispersibility can be obtained. 0.5 - 10 % of the weight is suitable for the addition to the black oxide obtained. When the amount of a sintering inhibitor is 0.5 or less % of the weight, the sintering prevention effectiveness is weak, and reduction is hard coming to progress and is not desirable when it is 10 % of the weight or more.

[0024] Moreover, also in the process of this invention, covering processing of a kind of water and inorganic oxides, such as silicon, aluminum, titanium, a zirconium, and tin, or an anhydrous and an inorganic oxide or the two sorts or more can be carried out for Fe₂TiO₄ above-mentioned Aigami's front face, or it is desirable to carry out covering processing of kinds, such as a silicone oil which is a hydrophobing agent, a silane coupling agent, and a titanium coupling agent, or the two sorts or more, and it can combine surface treatment processing of these various kinds with arbitration.

[0025] What is necessary is just to carry out by the well-known approach, when carrying out covering processing of the inorganic oxide hydrous [, such as the above-mentioned silicon, aluminum, titanium, a zirconium, and tin,] or anhydrous. Namely, what is necessary is just to process by adding water-soluble salts, such as silicon, aluminum, titanium, and a zirconium, more than a kind, neutralizing using alkali or an acid, and depositing the hydroxide of water or nothing, after slurring the obtained black multiple oxide with water and performing wet grinding for a water slurry using a pebble mill, a Sand grinder mill, attritor, etc. Moreover, what is necessary is just to process with a well-known wet method or dry process, also when carrying out hydrophobing processing using a hydrophobing agent. As a hydrophobing agent, coupling agents, such as silicone oils, such as a methylphenyl polysiloxane, dimethylsiloxane, H denaturation polysiloxane, and a fluorine denaturation polysiloxane, and a silane coupling agent, a titanium coupling agent, can be used, and these may be used together.

[0026] The low magnetism black pigment powder of this invention is effective also as a black pigment in a coating, a resin constituent, and the charge of makeup. It not only can use it as a common coating coloring agent, but as an object for coatings, it can use it as a coloring agent for heat-resistant coatings.

[0027] Moreover, as a resin constituent, it can be used also as a coloring agent of thermosetting resin, such as not only the coloring agent of general-purpose resin, such as vinyl chloride resin, polyethylene resin, and polypropylene resin, but an unsaturated polyester resin, an epoxy resin, polyurethane resin, etc.

[0028] Moreover, in the charge application of makeup, it can be used as a black pigment, such as finishing [foundation, eye shadow, and eyebrows].

[0029]

[Example] Although an example and the example of a comparison are shown below and this invention is explained to it, this does not limit this invention at all.

[0030] In addition, the particle size in the following examples and examples of a comparison is a pitch diameter for which it asked from the electron microscope photograph, and the magnetization value measured powder in the magnetic field of 5kOe(s) by Toei Industry VSM. whenever [moreover, / black] -- (L value) powder -- pressing -- the Suga Test Instruments Co., Ltd. make -- it measured by color circuit tester SC-2-CH. About glossiness, after having ground the obtained powder, having kneaded 5g of styrene-ized alkyd resins, and 0.5g of grinding articles by FUBA MARA, having coating-ized, applying to art paper with the 6-mil doctor blade and performing baking at 110 degrees C for 10 minutes, 20-degree glossiness of -20 " was measured with the Murakami color lab gloss meter.

[0031]

[Example 1] It adjusts [1.] in 150g /by using as titanium oxide the water titanium oxide slurry of specific-surface-area of 260m²/g obtained by the sulfuric-acid method, and pH is neutralized to 9 using 400g [/l.] caustic alkali of sodium. After 2-hour churning, the 200g [/l.] hydrochloric acid adjusted pH to 6, and filtration washing was performed. After having repulped the water titanium oxide which washed, using the 100g [/l.] ferric-chloride solution for the slurry after adjusting [g / / 100 / l.] as Fe₂O₃ as titanium oxide and carrying out 1 weight section addition to the titanium oxide 1 weight section,

the 200g [1.] caustic-alkali-of-sodium solution was dropped, pH of this slurry was adjusted to 7, and the iron hydroxide was covered on water and a titanium oxide front face.

[0032] After agitating for 1 hour, filtration and washing were performed and it dried at 110 degrees C. The dry matter was put into the porcelain crucible and the titanium oxide which performs baking at 900 degrees C with an electric furnace for 1 hour, and has Fe_2TiO_5 phase was compounded. Hydrogen gas and the mixed gas of carbon dioxide gas performed reduction for the titanium oxide which has Fe_2TiO_5 obtained phase after cooling at 500 degrees C for 5 hours, and black powder was obtained.

[0033] Drawing 1 is the X diffraction Fig. of the obtained product. As for the rutile type titanium dioxide and Peak B, the peak A in drawing shows Fe_2TiO_5 , as for Fe_2TiO_4 and Peak C. The rutile mold TiO_2 was made into MEIMPI-KU at the X diffraction, and the peak of Fe_2TiO_4 was included. That is, the X diffraction Fig. showed that the above-mentioned product was the mixed phase crystal constituted with the multiple oxide which uses TiO_2 (rutile type titanium dioxide) and Fe_2TiO_4 phase as a principal component. In addition, drawing 2 is the electron microscope photograph (x30,000) of the obtained black multiple oxide.

[0034]

[Examples 2-5, the examples 1-3 of a comparison] The amount, burning temperature, and reduced temperature of the iron hydroxide covered on the water and titanium oxide front face in an example 1 were changed as shown in Table 1, and the multiple oxide was compounded. The property of the obtained product is shown in Table 1.

[0035]
 [Table 1]

	Fe ₂ O ₃ 施理量 wt %	焼成温度 ℃	還元温度 ℃	還元時間 hrs	比表面積 m ² /g	粒径 μm	磁化値 emu/g	L 値	光沢度 %	色
実施例 1	100	900	500	5	6.7	0.22	6.5	9.1	46.9	黒色
実施例 2	100	1,050	500	8	4.2	0.38	2.5	8.7	55.2	黒色
実施例 3	100	750	500	5	8.6	0.18	8.9	9.7	43.3	黒色
実施例 4	100	800	400	5	7.1	0.20	7.1	10.2	45.5	黒色
実施例 5	55	800	500	5	6.5	0.20	4.9	12.2	49.1	黒色
比較例 1	55	900	350	5	7.4	0.17	0.9	27.5	59.2	褐色
比較例 2	20	900	500	5	7.1	0.21	1.4	30.2	56.2	灰色
比較例 3	200	900	500	5	6.3	0.25	1.3	15.7	57.3	黒褐色
参考例	—	—	—	—	7.5	0.20	82.2	9.4	29.3	黒色

[0036]

[Related Example(s)] It included in Table 1 by making the magnetite for toners into the example of reference. That is, though it is the pigment which does not use magnetite, in order to evaluate the point that the pigment property which is equal to magnetite was acquired, whenever it was black, the glossiness of a paint film was similarly measured about the magnetite currently used abundantly as an object for toners.

[0037] Even if it compared the black multiple oxide of this invention with the magnetite for toners

shown as an example of reference so that clearly from the result of Table 1, specific surface area, particle size, and whenever [black] were almost equivalent. Moreover, since the glossiness of a paint film is more remarkably [than the magnetite for toners] good, there is almost no magnetic condensation and dispersibility is considered to be good.

[0038]

[Example 6] 2.5% of the weight of the aluminum-sulfate solution was added as aluminum 2O₃ to the black oxide, having heated at 70 degrees C and agitating well, after repulping the black powder obtained in the example 1 to pure water and performing wet grinding in a sand mill. Then, the sodium-hydroxide solution was added, pH was adjusted to 7.5, and the aluminum hydroxide was covered. Filtration, washing, desiccation, and grinding were performed after 1-hour aging, and the aluminum₂O₃ covering black oxide was obtained.

[0039] When 20-degree glossiness of -20 " of the paint film using the obtained black oxide was measured, dispersibility was improved at 51.9%.

[0040]

[Example 7] an example 6 -- setting -- hexyl trimethoxysilane after covering an aluminum hydroxide -- a black oxide -- receiving -- 5 % of the weight -- it added, and it hydrolyzed underwater, the silane coupling agent was processed, filtration, washing, desiccation, and grinding were performed, and the silane coupling agent covering black oxide was obtained.

[0041] When 20-degree glossiness of -20 " of the paint film using the obtained black oxide was measured, it is 53.7% and dispersibility was improved further.

[0042]

[Effect of the Invention] Since the black multiple oxides of this invention are insurance and a harmless black pigment and it is low magnetism, there is almost no magnetic condensation, and since distribution is good, it is suitable as coloring agents, such as a toner for electrostatic development, a coating, resin, and a charge of makeup.

[0043]

[Translation done.]

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